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Strength, Fracture Toughness, Fatigue, and Standardization Issues of Free-Standing Thermal Barrier Coatings

Sung R. Choi
Ohio Aerospace Institute, Brook Park, Ohio

Dongming Zhu
U.S. Army Research Laboratory, Glenn Research Center, Cleveland, Ohio

Robert A. Miller
Glenn Research Center, Cleveland, Ohio

National Aeronautics and
Space Administration

Glenn Research Center

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Acknowledgments

This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

This report contains preliminary findings, subject to revision as analysis proceeds.

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NASA Glenn Research Center, Cleveland, OH

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Abstract

**Strength, Fracture Toughness, Fatigue, and Standardization Issues of Free-Standing
Plasma-Sprayed Thermal Barrier Coatings**

Sung R. Choi, Dongming Zhu, and Robert A. Miller
NASA Glenn Research Center, Cleveland, OH 44135

Strength, fracture toughness and fatigue behavior of free-standing thick thermal barrier coatings of plasma-sprayed ZrO_2-8wt% Y_2O_3 were determined at ambient and elevated temperatures in an attempt to establish a database for design. Strength, in conjunction with deformation (stress-strain behavior), was evaluated in tension (uniaxial and trans-thickness), compression, and uniaxial and biaxial flexure; fracture toughness was determined in various load conditions including mode I, mode II, and mixed modes I and II; fatigue or slow crack growth behavior was estimated in cyclic tension and dynamic flexure loading. Effect of sintering was quantified through approaches using strength, fracture toughness and modulus (constitutive relations) measurements. Standardization issues on test methodology also was presented with a special regard to material's unique constitutive relations.
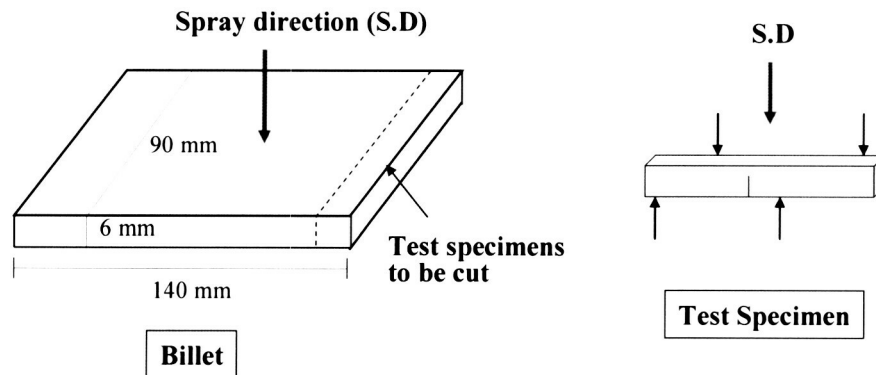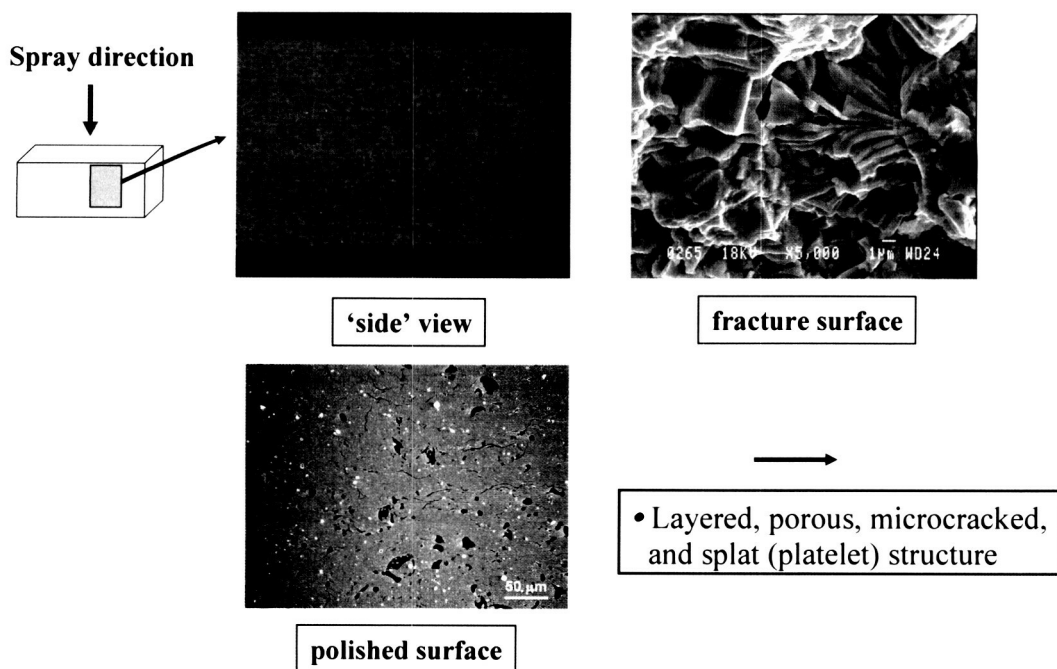
Contents

I. Backgrounds

- **Thermal Barrier coatings (TBCs), ZrO_2-8 wt% Y_2O_3 – important coating materials due to low thermal conductivity, high thermal expansivity, and unique microstructure**

- **Somewhat anisotropic nature of porosity, microcracks and splat structure - a challenge in routine mechanical testing and data interpretation**

- **Mechanical testing for TBCs performed to characterize strength, fracture toughness, fatigue, and deformation, and also to establish database**

- **Results of mechanical testing presented and discussed, and related issues discussed**

II. Material Processing

- **ZrO$_2$-8 wt% Y$_2$O$_3$ powder** with an average particle size of 60 μm
- Plasma sprayed on a steel or graphite substrate
- SULZER-METCO ATC-1 plasma coating system with a 6-axes industrial robot used
- **Free standing** TBC billets fabricated
- Test specimens machined from billets with appropriate configurations
- Typical billets:

Spray direction (S.D)

90 mm

6 mm

140 mm

Test specimens to be cut

Billet

S.D

Test Specimen

Unique Microstructure of TBCs

Spray direction

'side' view

fracture surface

0265 18KU X5,000 1μm WD24

polished surface

60 μm

- Layered, porous, microcracked, and splat (platelet) structure

III. Strength Testing

Types of Testing/Test Specimens/Orientations

Spray direction (S.D)

90 mm

6 mm

140 mm

Test specimens to be cut

Billet

Uniaxial tension   **Trans-thickness tension**   **Uniaxial compression**

Uniaxial flexure (four-point)   **Biaxial flexure (ring-on-ring)**

↓ indicates spray direction
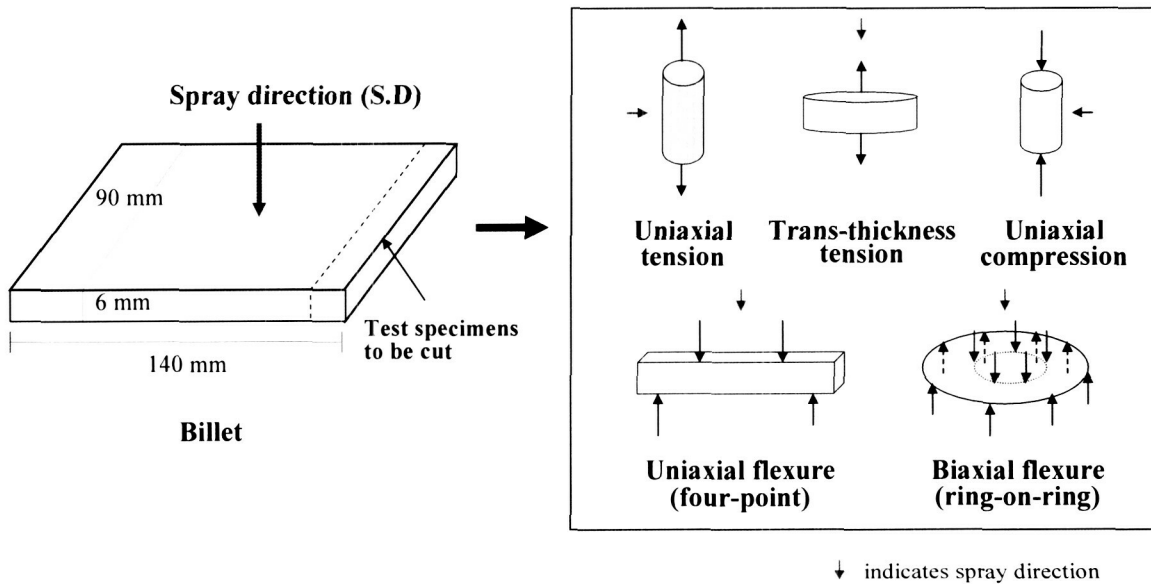
Test Matrix (strength)

Type of tests	Specimen geometry	No. of test specimens	Direction of fracture*
Uniaxial tension	15mm x 5mm⬦	10	P
Trans-thickness Tension	15 mm x 3 mm (t) (diameter x thick.)	10	N
Uniaxial compression	10mm x 5mm⬦	10	P
Uniaxial flexure (four-point)	3mm x 4mm x 25mm [10/20 mm spans]	30	P
Biaxial flexure (ring-on-ring)	25mm x 3mm (t) [11/22 mm rings]	10	P

Uniaxial tension   **Trans-thickness tension**   **Uniaxial compression**

Uniaxial flexure (four-pt.)   **Biaxial flexure (ring-on-ring)**

↓ indicates spray direction
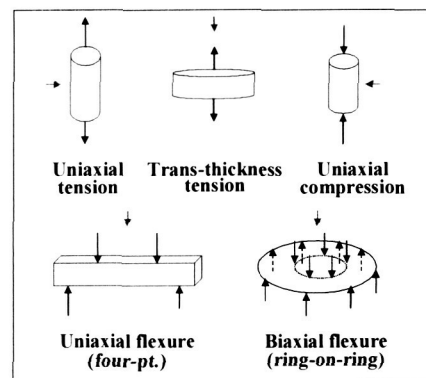
* indicates the direction of fracture w.r.t plasma-spray direction.
Test temperature: ambient temperature in air.

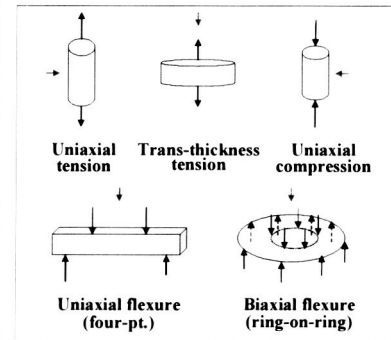# Experimental Results (strength)

Type of tests	No. of test specimens valid	Direction*	Average strength (MPa)#	Weibull modulus
Uniaxial tension	3	P	15(1)	-
Trans-thickness Tension	10	N	11(1)	13
Uniaxial compression	10	P	300(77)	4
Uniaxial flexure (four-point)	30	P	33(7)	6
Biaxial flexure (ring-on-ring)	10	P	40(4)	12

↓ indicates spray direction

* indicates fracture direction w.r.t plasma-spray direction:
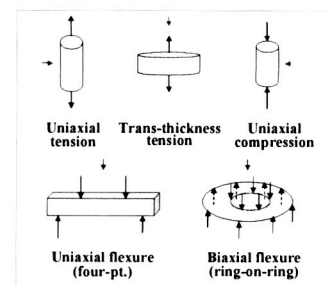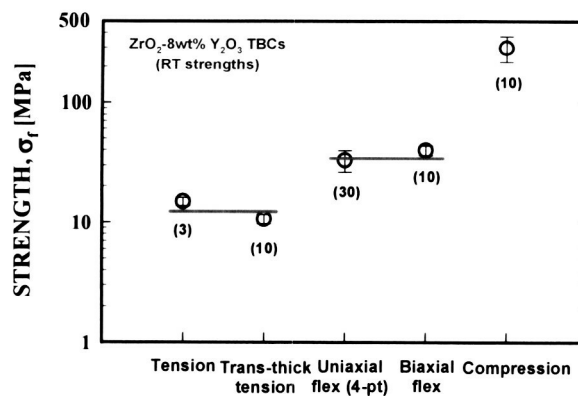 N: normal; P: parallel
\# represents ±1.0 standard deviation

→ A basic assumption in strength calculation: a continuum mechanics (isotropic and linear-elastic)

σ_f = 10-15 MPa in tension
= 30-40 MPa in flexure
= 300 MPa in compression

Choi, Zhu, and Miller ('98,'99,'00,'01)

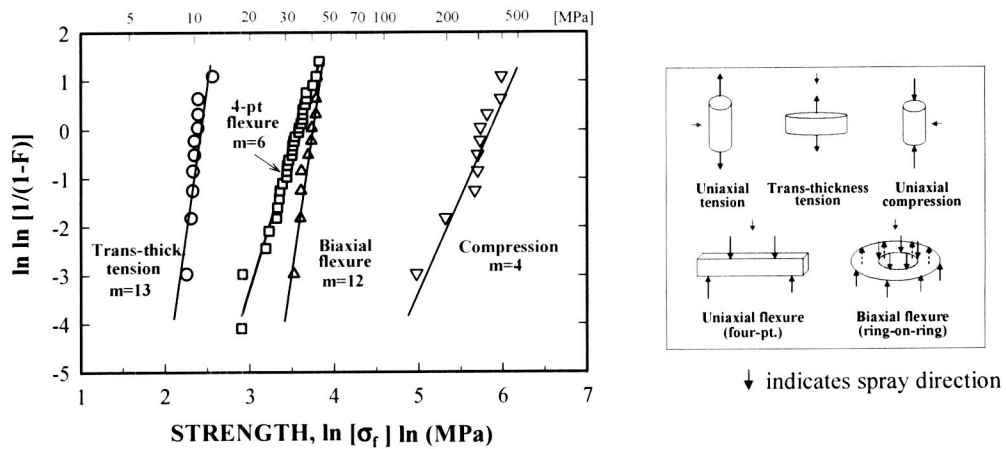# Experimental Results (strength)

Strength vs Type of Tests

↓ indicates spray direction

→ σ_f = 10-15 MPa in tension
= 30-40 MPa in flexure
= 300 MPa in compression

The numbers indicates the number of specimens tested valid

Experimental Results (strength)
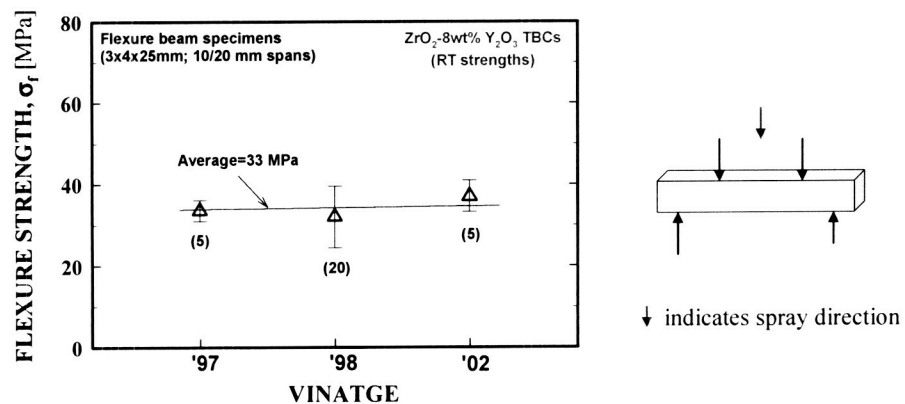
Weibull Strength Distributions

[MPa] scale: 5, 10, 20, 30, 50 70 100, 200, 500

y-axis: ln ln [1/(1-F)]

4-pt flexure m=6

Trans-thick tension m=13

Biaxial flexure m=12

Compression m=4

STRENGTH, ln [σf] ln (MPa)

Uniaxial tension | Trans-thickness tension | Uniaxial compression

Uniaxial flexure (four-pt.) | Biaxial flexure (ring-on-ring)

↓ indicates spray direction

- **Weibull moduli of m=5-15, a typical range for many commercial or in-house (dense) monolithic ceramics**

Choi, Zhu, and Miller ('98,'99,'00,'01)

Experimental Results (strength)

Flexure Strength *vs* Vintage

FLEXURE STRENGTH, σ_f [MPa]

Flexure beam specimens (3x4x25mm; 10/20 mm spans)

ZrO_2-8wt% Y_2O_3 TBCs (RT strengths)

Average=33 MPa

(5) (20) (5)

VINATGE: '97 '98 '02

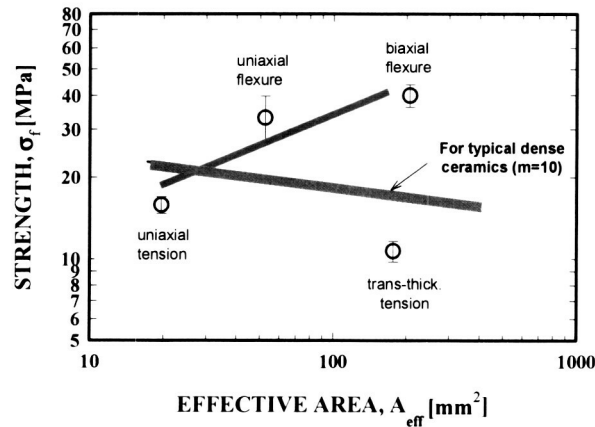↓ indicates spray direction

- **Flexure strength – less influence by vintage, indicating consistency in plasma-spray processing over the years**

Choi, Zhu, and Miller ('98,'99,'03)

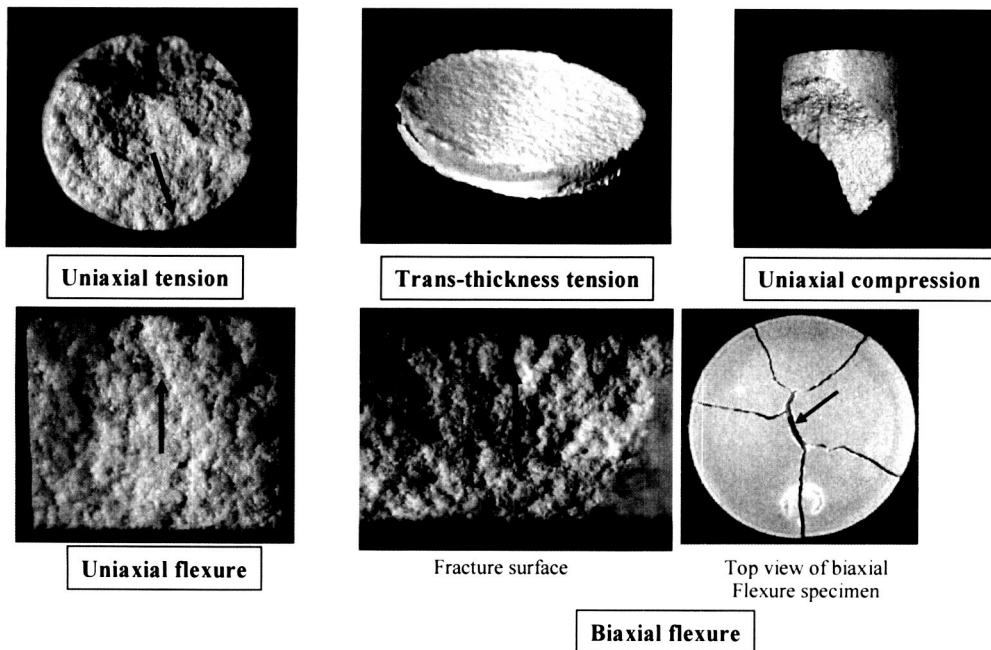The numbers indicates the number of specimens tested valid

Strength vs. Effective Area – Size Effect

Strength-Effective Area (Weibull PIA model)

- **No reasonable agreement** in size effect between data and Weibull analysis (e.g., PIA); inconsistency in flaw populations (?)


Fractography (strength)

| Uniaxial tension | Trans-thickness tension | Uniaxial compression |

Uniaxial flexure

Fracture surface   Top view of biaxial Flexure specimen

Biaxial flexure

- **Very difficult to locate fracture origins and to analyze their nature**

Fractography – A Great Challenge

Four-point flexure

TBCs

Dense Ceramics

ZrO$_2$-8wt% Y$_2$O$_3$

Toughened Si$_3$N$_4$

ZrO$_2$-10 mol% Y$_2$O$_3$
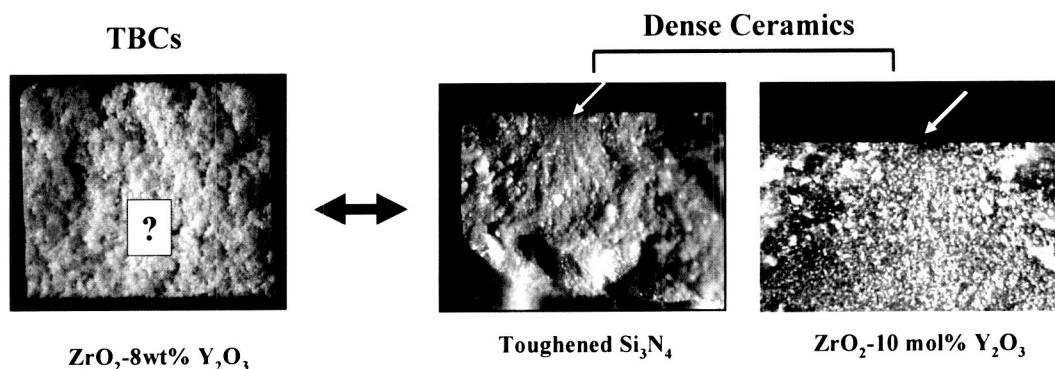
Fracture mirror size (r$_m$):
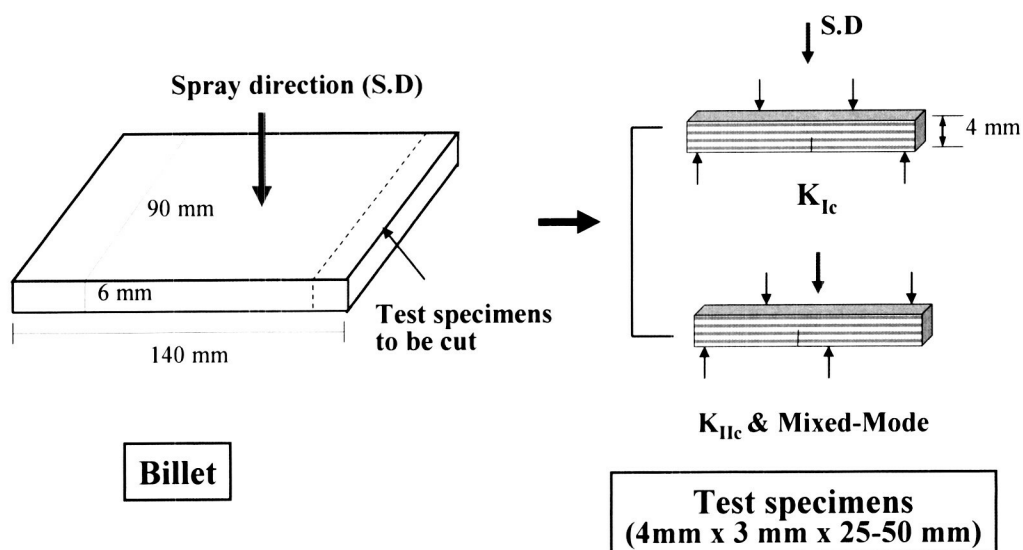- TBCs - 3-40 mm (estimated)
- Dense ceramics – 50-500 μm

$$\sigma_f r_m^{1/2} = A$$

• Big mirror size & porous/microcracked nature of TBCs
→ An enormous challenge in fractogrphy

Choi, Zhu, and Miller ('00,'01)
Choi ('02); Choi and Narottam ('02)

IV. Fracture Toughness Testing
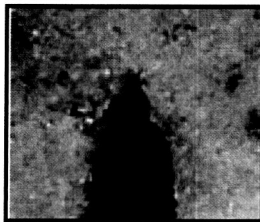## (Mode I, Mode II and Mixed Mode)

Types of Testing/Test Specimens/Orientations

Spray direction (S.D)

90 mm

6 mm

140 mm

Test specimens
to be cut

Billet

S.D

4 mm

K$_{Ic}$

K$_{IIc}$ & Mixed-Mode
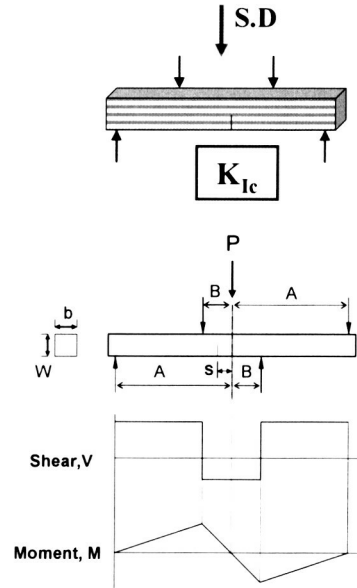
**Test specimens
(4mm x 3 mm x 25-50 mm)**

Experimental (fracture toughness)
(Mode I, Mode II and Mixed Mode)

Types & Procedures

- **Sharp precracks generated**
  - Single edge v-notched beam (**SEVNB**) method: Saw-notched → a sharp V-notch generated with a razor blade with diamond paste, $a/W \approx 0.5$
- **Test fixture configurations**
  - A/B=10/5 (typical); s=0-3.6 mm in mixed mode
  - 10/20 or 20/40 mm spans in K_{Ic}
- **Test temperatures**
  - 25 and 1316 °C in air
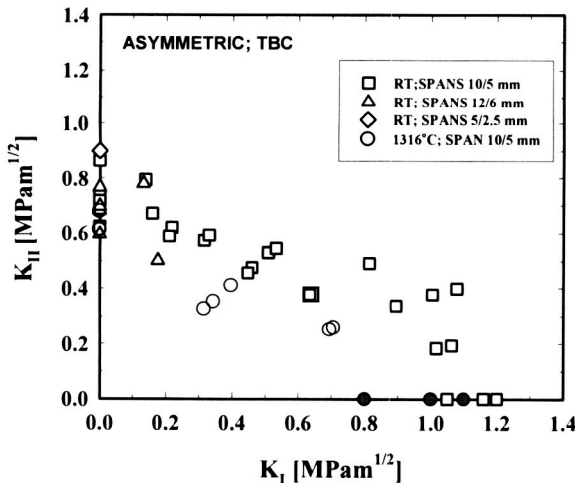- **Number of test specimens**: typically ≥4

A typical, sharp SEVNB precrack (root radius ≤30 μm)

K_{IIc} & Mixed-Mode -asymmetric flexure-

Experimental Results (fracture toughness)

- ## Mode I, Mode II, and Mixed Mode (25 and 1316 °C)

ASYMMETRIC; TBC

☐ RT;SPANS 10/5 mm
△ RT; SPANS 12/6 mm
◇ RT; SPANS 5/2.5 mm
○ 1316°C; SPAN 10/5 mm

K_{II} [MPa$m^{1/2}$]

K_{I} [MPa$m^{1/2}$]

Test Temp(°C)	No. of specimens used	K_{Ic} (MPa√m)	K_{IIc} (MPa√m)
25	4 in K_{Ic} 9 in K_{IIc}	1.15(0.07)	0.73(0.10)
1316	4 each	0.98(0.13)	0.65(0.04)

- $K_{Ic} > K_{IIc} \rightarrow K_{IIc}/K_{Ic}$=0.64 & 0.66 (at 25 & 1316 °C)
- K_{Ic} and K_{IIc} at 25 °C ≥ K_{IC} and K_{IIc} at 1316 °C
- **Elliptical relation between K_{I} and K_{II}**
- Test spans **independent**

Choi, Zhu, and Miller ('03)

Experimental Results (fracture toughness)

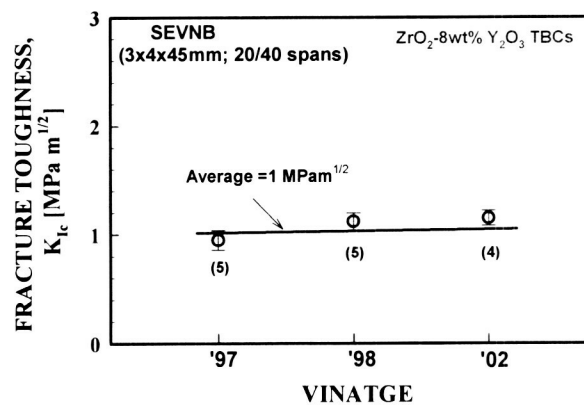- ## Fracture Toughness vs. Temperature
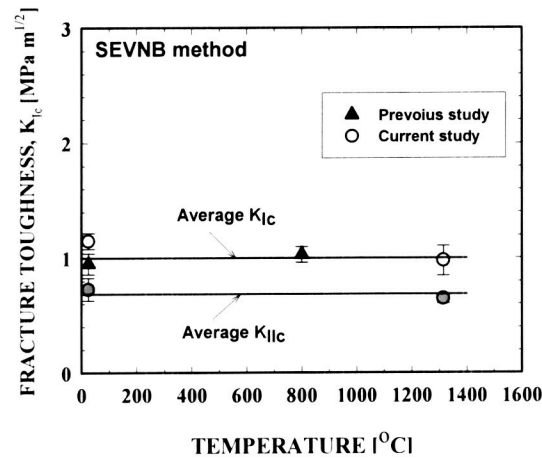
- **Temperature insensitive in K_{Ic} and K_{IIc}**
  $\rightarrow K_{Ic} \approx 1$ and $K_{IIc} \approx 0.65$ MPa$\sqrt{m}$
- $K_{IIc}/K_{IC} \approx 0.65$

Choi, Zhu, and Miller ('98,'03)

Experimental Results (fracture toughness)
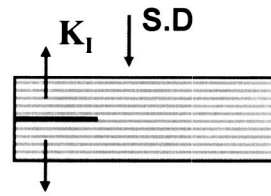
- ## Fracture Toughness (RT) vs. Vintage

- **Fracture toughness (K_{Ic}) – less influence by vintage (similar to strength), indicating consistency in plasma-spray processing over the years**
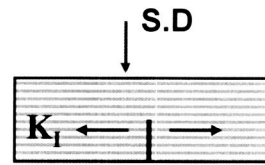
Choi, Zhu, and Miller ('98,'03)

Experimental Results (fracture toughness)

Fracture Toughness vs. Orientation

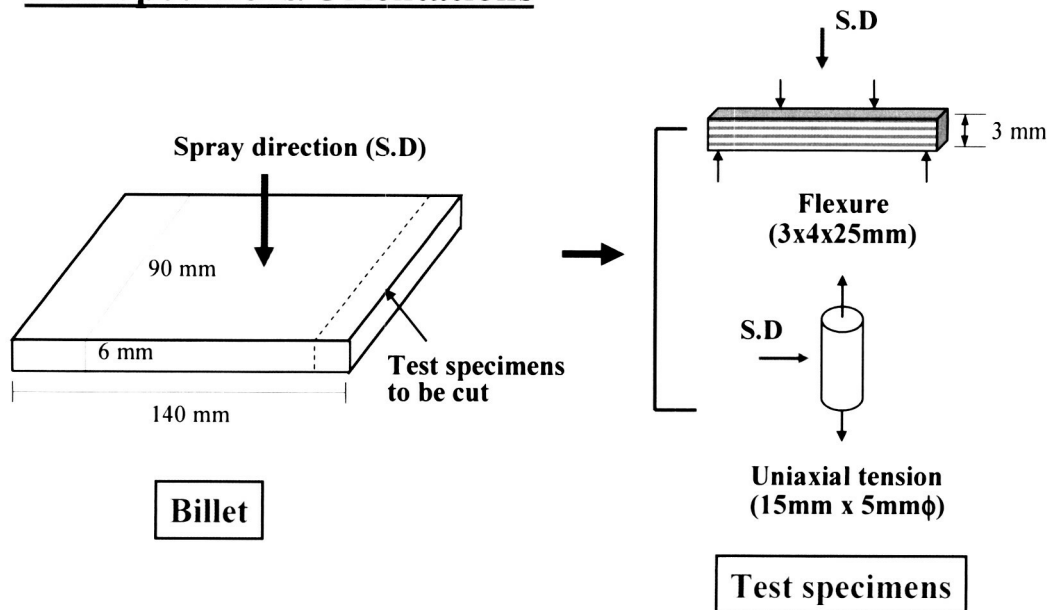DCB specimens   SEVNB specimens

Direction of crack	Fracture Toughness K_{IC} (MPa√m)	Method
Parallel to plasma spray direction	1.15±0.07	SEVNB (regular method)
Normal to plasma spray direction	1.04±0.05	DCB (Double Cantilever Beam)

⟶ • No significant difference in K_{Ic}-- Little directionality effect on K_{Ic}

Choi, Zhu, and Miller ('98,'03)

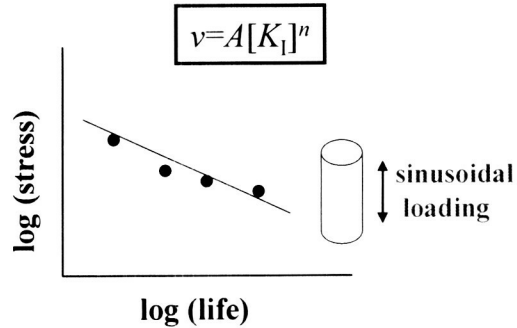# V. Fatigue/Slow Crack Growth

Test Specimens/Orientations

Spray direction (S.D)

90 mm

6 mm

140 mm

Test specimens to be cut

Billet

S.D

3 mm

Flexure (3x4x25mm)

S.D

Uniaxial tension (15mm x 5mmφ)

Test specimens

Experimental (fatigue)

Test Types and Conditions

- **Dynamic fatigue (ASTM C1425)**
  800 °C in air; 3 test rates in flexure
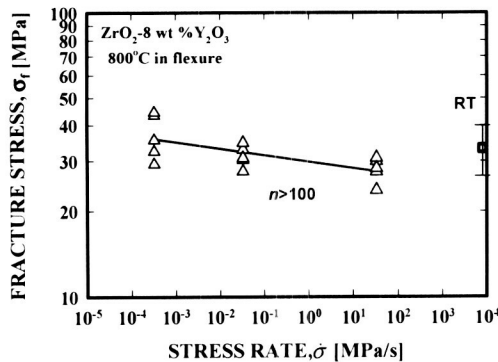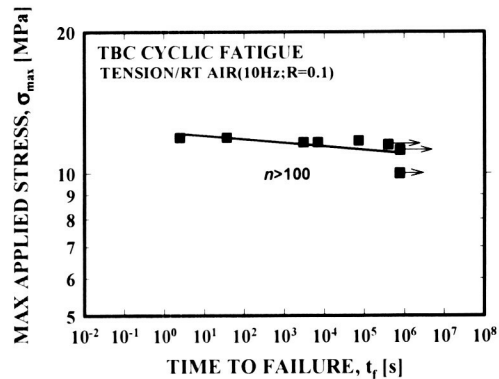- **Tensile cyclic fatigue**
  RT in air; sinusoidal; R= 0.1; f=10 Hz
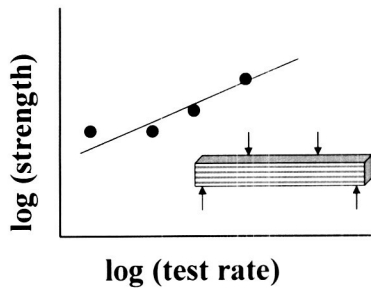
$$v=A[K_I]^n$$

Dynamic fatigue

Tensile cyclic fatigue

Experimental Results (fatigue/SCG)

Dynamic fatigue

Tensile cyclic fatigue
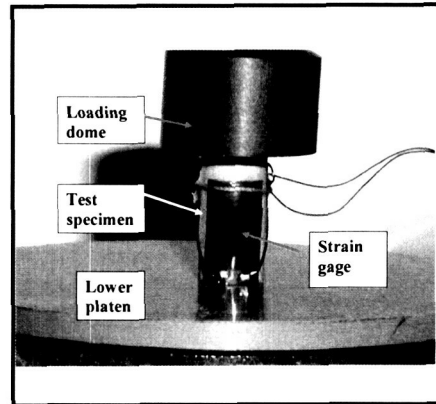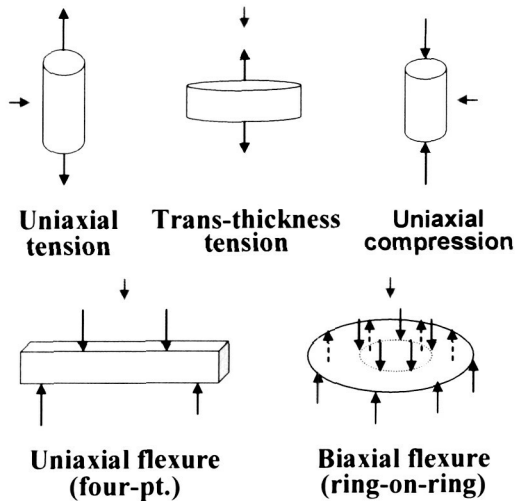

$$v=A[K_I]^n$$

→ - Slow crack growth (SCG) parameter n:
  $n > 100$ in both dynamic and tensile cyclic fatigue –
  Little susceptibility to SCG (fatigue)

Choi, Zhu, and Miller ('98,'99,'01)

VI. Deformation (Stress-Strain) Behavior

5 Specimen/Loading Conditions Considered

Uniaxial tension   Trans-thickness tension   Uniaxial compression

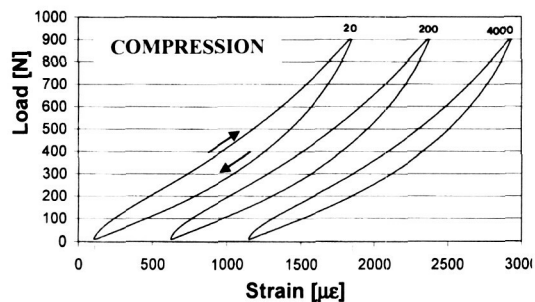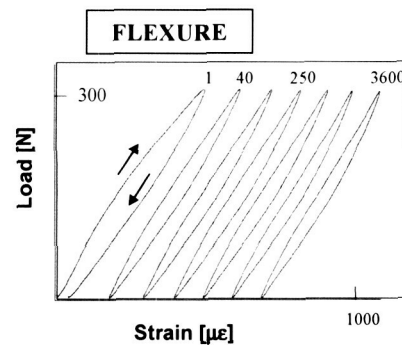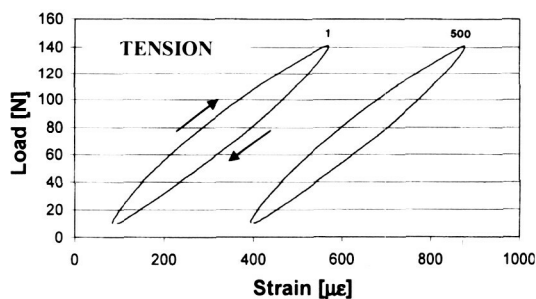Uniaxial flexure (four-pt.)   Biaxial flexure (ring-on-ring)

Test Setup (strain gaging)

↓ indicates spray direction

Experimental Results (deformation)

Typical Load-Strain Curves
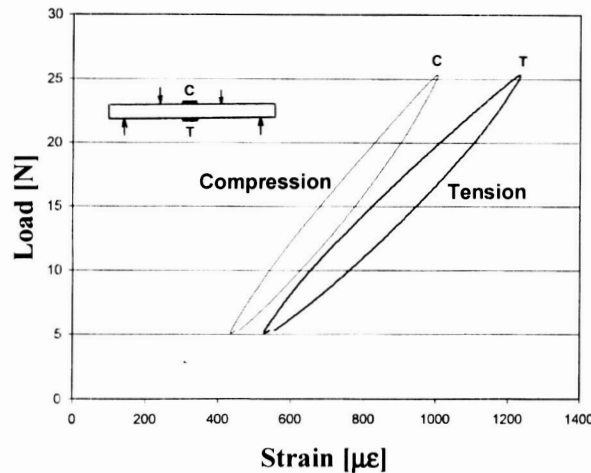

FLEXURE

TENSION

COMPRESSION

- **Non-linearity with hysteresis but elastic
-desirable in TBCs but difficulty in analysis**
- Independent of the <u>number of cycles</u> and <u>test rate (not-viscoelastic)</u>

Choi, Zhu, and Miller ('00,'01)

Experimental Results (deformation)
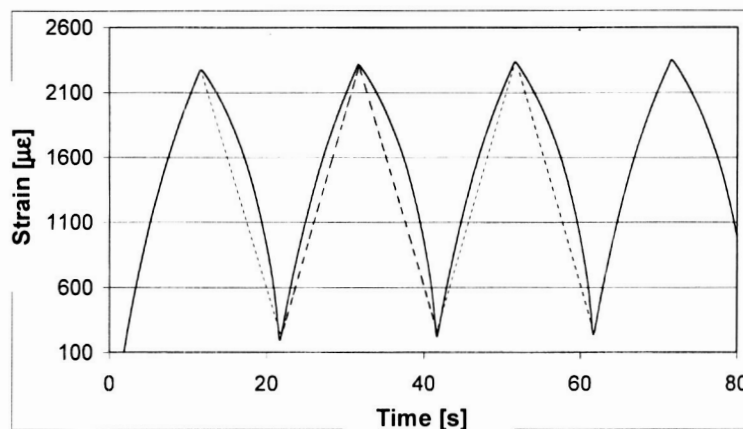
Four-Point Flexure

- **Different response of strain** in compression and tension
- A possible **neutral axis shift** due to different elastic modulus
- Flexure stress calculation – complex

Choi, Zhu, and Miller (''01)

Experimental Results (deformation)

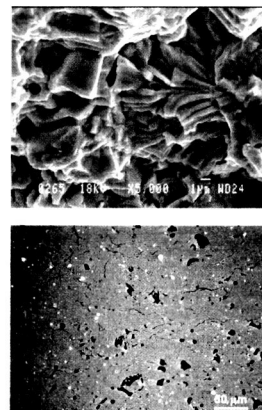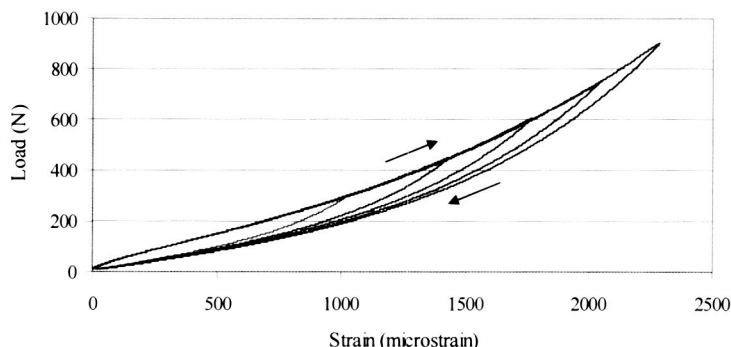## Response of Output Wave Form to Cyclic Compression Loading

The output wave form - **distorted** from the input triangular wave form

Choi, Zhu, and Miller ('01)

Deformation (Stress-Strain) Behavior

What is the cause of nonlinearity and hysteresis?

Major reason – '**loosely' connected open structure** due to pores and microcracks
- Internal friction and densification
- Still overall elastic behavior

Eldridge, Morscher, and Choi ('02)

Deformation (Stress-Strain) Behavior

'Loosely-Connected Open' Structure – Poisson's Response
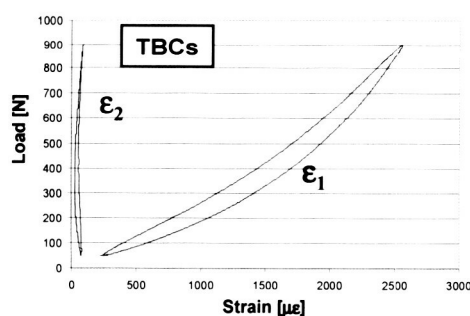
Exhibits no or little change in lateral strain: Poisson's ratio not well defined

Exhibits a linearly increasing lateral strain: Poisson's ratio well defined

Poisson's ratio
$v=|\varepsilon_2/\varepsilon_1|$

Open, loose structure
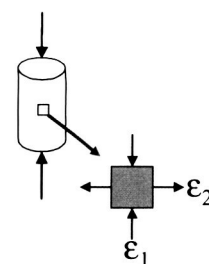
Dense structure

Experimental Results (deformation)

Sandstone – Another Example of Open Structure
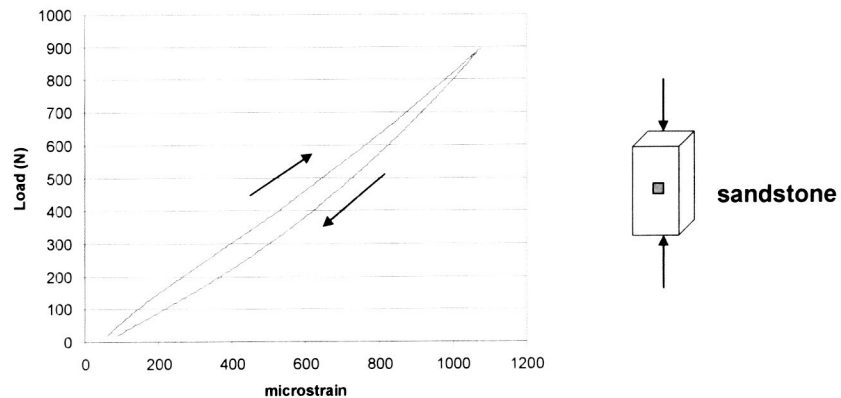
Open, loose structure: non-linearity with hysteresis
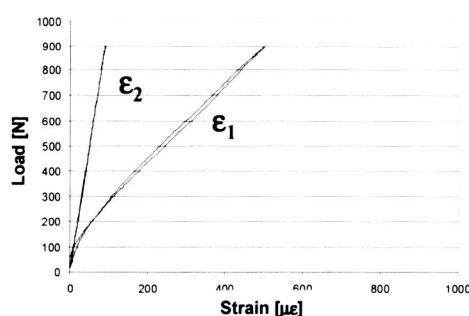-- Similarity to TBCs

VII. Sintering – A Changer of Structure

Sintering conditions:

- Temperature/environment: 1316 °C/air
- Annealing time: 0, 5, 20, 100, and 500 h
- Determine as a function of anneal time:
  - Elastic modulus
  - Fracture toughness (K_{Ic})
  - Flexure strength
  - Thermal conductivity

| Elastic modulus | Flex strength | Fracture toughness |
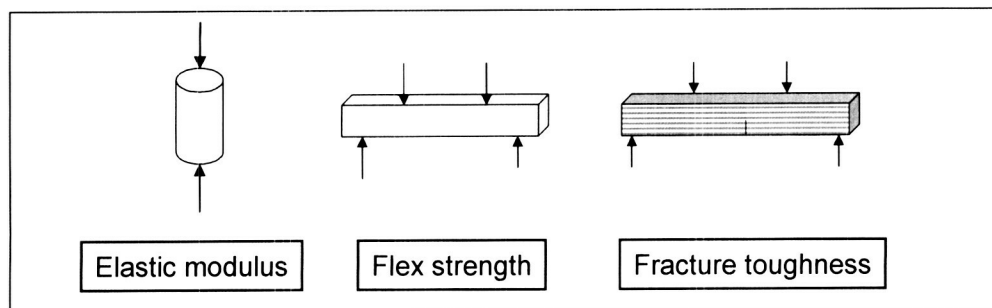
Experimental Results (sintering)

Elastic Modulus
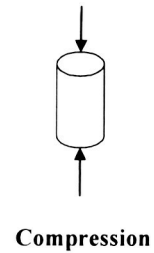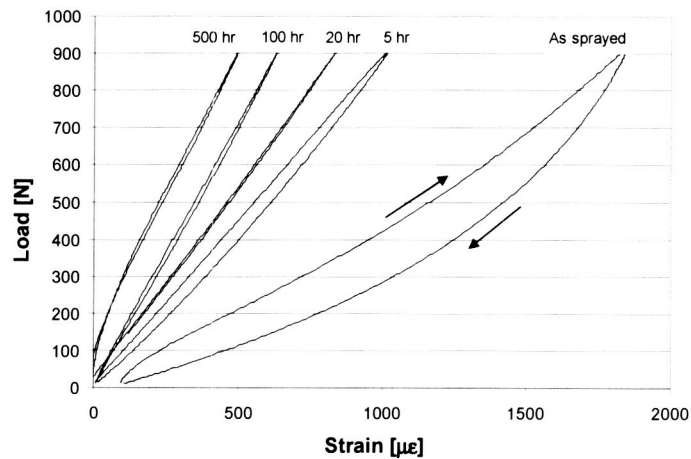
- Slope (elastic modulus) increases with anneal time
- Linearity increases with anneal time
- Hysteresis decreases with anneal time

⟶ ☐ Implies a change of microstructure from 'loosely' connected to 'closely' connected

Experimental Results (sintering)

Well-Developed Poisson's (Lateral Strain) Response
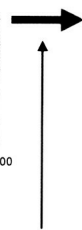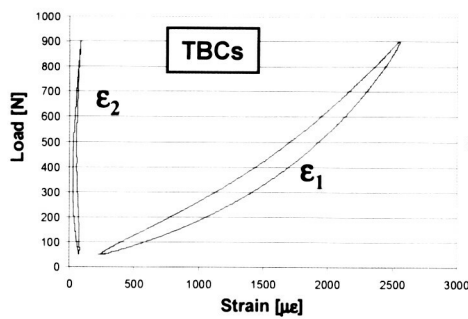
500 h annealing
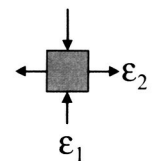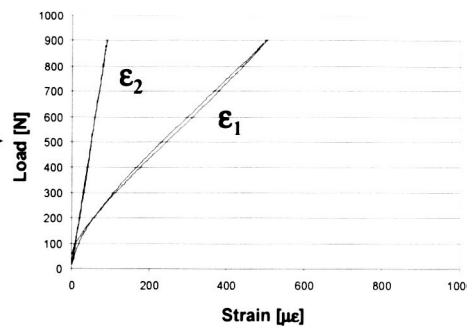
Poisson's ratio
$v = |\varepsilon_1 / \varepsilon_2|$

Open structure → More closely-connected structure

Experimental Results (sintering)

Microstructure
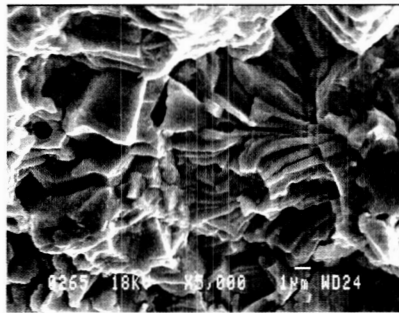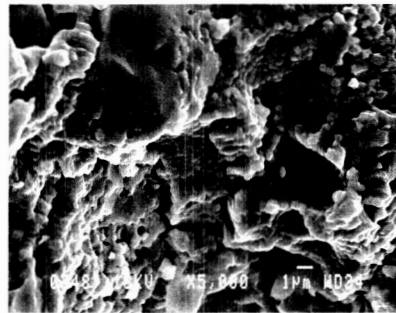
| As-sprayed | 100 h annealed |

- **As-sprayed** - Large amounts of microcracks and pores **with a unique** platelet (splat) structure presented
- **100 h annealing** - Increased grain growth **at longer annealing time**

Experimental Results (sintering)

- **Summary on elastic modulus, flexure strength, fracture toughness and thermal conductivity**
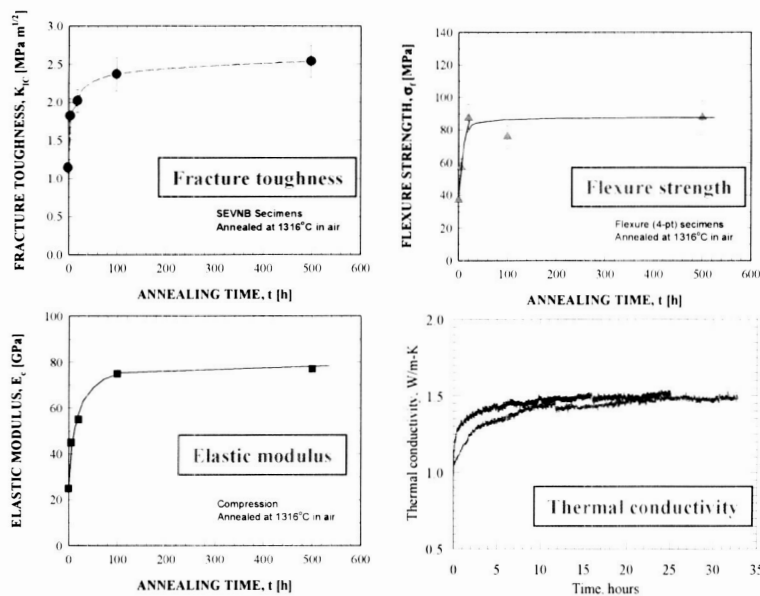
⟶ **Properties change exponentially with sintering time**   Choi, Zhu, and Miller ('03)

Standardization Issues

- **The most hindering factor** in establishing test methods for as-sprayed TBCs: **non-linearity & hysteresis** in the constitutive relations
  - <u>Flexure testing</u> (uniaxial and biaxial) maybe inappropriate due to difference in modulus between tension and compression
  - <u>Poisson's ratio</u> not well-defined
  - <u>Impulse excitation technique</u> maybe inappropriate

- <u>Pure tension and compression testing</u> – impose less problems

- <u>Fracture toughness testing</u> – maybe OK in view of low fracture loads

- <u>Fractography</u> – challenging

- <u>Properties change with sintering/service conditions</u>
  - requires to evaluate based on sinter/service conditions

Summary

- <u>Strength:</u>
  tension: 10-15 MPa; flexure: 30-40 MPa; compression: 300 MPa
  Weibull modulus: 5-15
- <u>Fatigue/Slow Crack Growth:</u>
  SCG parameter n>100
- <u>Fracture Toughness:</u>
  K_{Ic}=1.0 MPa$\sqrt{m}$ up to 1316 °C
  K_{IIc}=0.7 MPa$\sqrt{m}$ up to 1316 °C
- <u>Deformation:</u>
  nonlinear elasticity with hysteresis;
  imposes problems in continuum approach (*test standards*)
- <u>Sintering:</u>
  significant influence - a changer of most properties!

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Sung R. Choi, Dongming Zhu, and Robert A. Miller

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Strength, fracture toughness and fatigue behavior of free-standing thick thermal barrier coatings of plasma-sprayed ZrO_2-8wt % Y_2O_3 were determined at ambient and elevated temperatures in an attempt to establish a database for design. Strength, in conjunction with deformation (stress-strain behavior), was evaluated in tension (uniaxial and trans-thickness), compression, and uniaxial and biaxial flexure; fracture toughness was determined in various load conditions including mode I, mode II, and mixed modes I and II; fatigue or slow crack growth behavior was estimated in cyclic tension and dynamic flexure loading. Effect of sintering was quantified through approaches using strength, fracture toughness, and modulus (constitutive relations) measurements. Standardization issues on test methodology also was presented with a special regard to material's unique constitutive relations.

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